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First Far-IR Microscopy at BL1.4.3

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Extending the reach of synchrotron infrared microscopy to longer wavelengths will allow users to measure lower frequency vibrational modes. Presently, the Nic-Plan IR microscope on Beamline 1.4.3 has a MCT-A detector with a low frequency cut-off of ~650 cm^{-1}. The KBr beamsplitter will in principle allow measurements down to about 400 cm^{-1}. To measure lower frequencies will require a different beamsplitter and a different detector.

To test the longer wavelength capabilities of the beamline and the instrumentation, we purchased a solid silicon beamsplitter (nominal rage 50 – 700 cm^{-1}), and we manually added a flat mirror inside the Nic-Plan's detector compartment to direct the light from the sample out the side of the microscope. This light was then focused using an off-axis parabolic mirror onto the entrance cone of a liquid helium cooled silicon bolometer. This Infrared Laboratories bolometer was used with its 700 cm^{-1} cut-on filter, and a Stanford Research low-noise amplifier to make the signal levels appropriate for the Nicolet ADC input. This detected IR signal was sent back into the Nicolet spectrometer for interferrogram acquisition and subsequent Fourier transform processing by the OMNIC software. The side of the microscope cover and the parabolic mirror were located in air for these initial tests, so the far-IR water vapor spectrum will be a limitation in performance. In the future we will enclose and purge this part of the optical path.

The front end of beamline 1.4 is not well-suited for collecting far-IR radiation as the vertical opening angle is limited to 10 milliradians by the 1 cm gap between the electron chamber and the antichamber – which means we are already starting to cut into the collected intensity at a couple microns wavelengths. By 100 microns (100 cm^{-1}), we are loosing a factor of ~5 due to this vertical collection restriction.

The measured raw single beam far-IR spectrum is shown in Figure 1. It was obtained using the synchrotron source in reflection off a gold-coated microscope slide and the 15x objective, with 128 scans, 4 cm^{-1} resolution, a scanning mirror velocity of 1.266 cm/sec, 20 times amplified with 100 Hz roll-on and 10 KHz roll-off filters. 100% signal to noise lines show 1% RMS noise from 220 to 675 cm^{-1} and usable signal to about 200 cm^{-1}, with the water vapor absorption locations degrading the signal to noise at each dip around this low frequency.

To test the spatial resolution of the focused synchrotron spot in the microscope and determine if we are indeed maintaining diffraction-limited performance into the far-IR

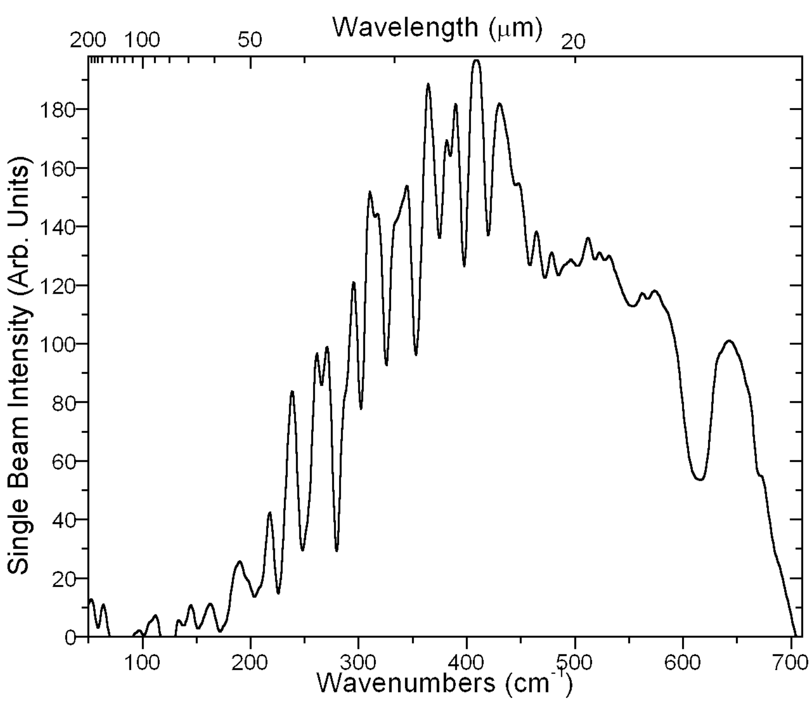

Figure 1. Single beam far-IR intensity measurement of the synchrotron source through the BL1.4.3 Nic-Plan IR microscope and using a Si beamsplitter and LHe cooled Si bolometer detector. The series of absorption bands below ~425 cm-1 are due to water vapor in the air. These will be reduced significantly with the addition of a purge box for the external detector optics.

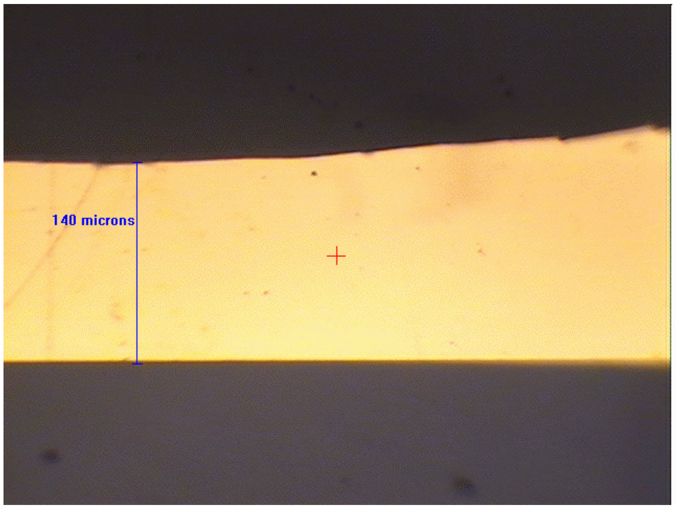

Figure 2. Photomicrograph of the gold strip on glass used to test the spatial resolution of the synchrotron beam as a function of wavelength.

a line scan across a test sample was performed. The test sample was a sharp edge of gold evaporated onto a glass slide. As shown in Figure 2, the gold test pattern had a 140 micron wide strip of gold, and a line scan with 5 micron steps was obtained from the glass, onto the gold, and then back onto the glass again.

The measured intensity was integrated over numerous ~50 cm^{-1} wide regions wherever there was significant intensity in the spectra as a function of distance across the line map. Several representative intensity versus position graphs are plotted in Figure 3. One can immediately recognize that the sharpness of this intensity step changes as a function of wavelength with short wavelengths showing a sharper rise than longer wavelengths, as expected. The solid lines in Figure 3 are fits to the data at each wavelength shown, and these fits allow us to determine a spatial resolution for each wavelength. The measured resolution is plotted as a function of wavelength in the inset of Figure 3, with the dashed line showing that the resolution follows a linear relation with wavelength with a slope of 0.78 – meaning the spot size is 0.78 λ. The error bars are large at the longest wavelengths as these farthest-IR signals were quite low.

The next steps are to make this far-IR mode of operation easier to set up, and maintaining the atmosphere around the beam well purged. These goals will require a mechanism for reproducibly inserting and removing a flat mirror into the Nic-Plan microscope for directing the beam to an external detector, and constructing and installing a sealed, purged optics box for transporting and focusing the beam onto the detector.

This new far-IR capability is important for matching astronomical observations, studying low frequency metallo-organic modes, surface electro-chemical products, etc.

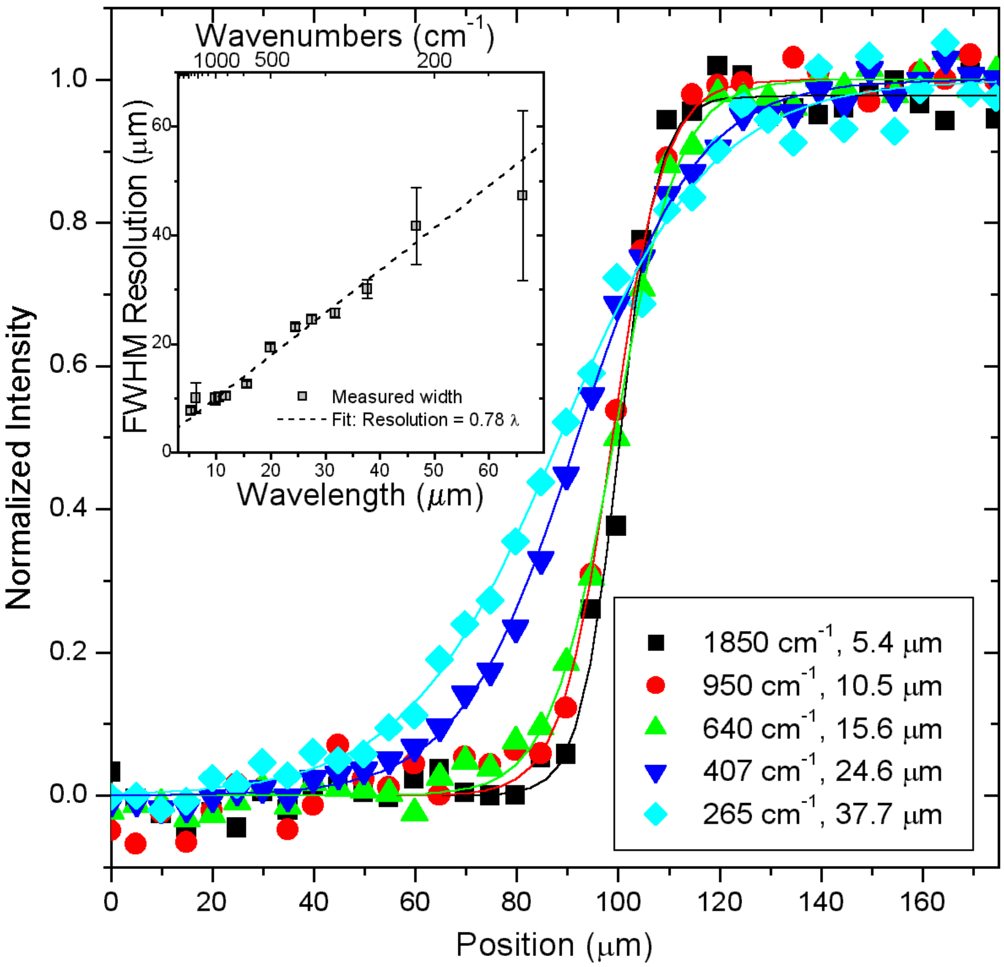

Figure 3. Plot of the intensity as a function of position for five representative wavelengths. The sharpness of the measured edge allows the determination of resolution. The measured resolution as a function of wavelength is shown in the inset.